

PROJECT NUMBER : 1752  
PROJECT TITLE : Molecular Structure Determination and Materials Evaluation  
PROJECT LEADER : G. Vilcins  
PERIOD COVERED : December, 1990

#### I. NMR ANALYSIS OF FLAVORS

- A. Objective: To use NMR to determine the structure and purity of novel flavor release compounds.
- B. Results: Several compounds in a series of alkyl pyrazines and pyridines were analyzed by  $^1\text{H}$  NMR. NMR was used to determine the positional isomers and confirm the overall structures. Two isomers of cyclopropylpyrazine and cyclopropylpyridine were analyzed by NMR. Several menthyl carbonates of methyl- $\alpha$ -D-glucopyranoside were analyzed by  $^1\text{H}$  and  $^{13}\text{C}$  NMR in order to determine the structure and purity.
- C. Conclusions: NMR is the method of choice for these types of analyses. The overall purity of these compounds were better than 95% in most cases.
- D. Plans: NMR will continue to support to the flavor-release and pyrolysis programs.
- E. Reference:

Bassfield, R., PM Notebook #7398, p. 176.

#### II. $^{13}\text{C}$ CPMAS NMR OF CALCIUM HYDROXIDE TREATED EXPANDED TOBACCO

- A. Objective: Determine the degree of esterification of pectin isolated from expanded tobacco and the effect of calcium hydroxide treatment on expanded tobacco and the isolated tobacco pectin.
- B. Results: The degree of methyl esterification of the pectin in expanded tobacco was found not to be significantly affected by calcium hydroxide treatment. Calcium ion binding to pectin carboxyl groups was indicated by a downfield shift of some of the carboxyl resonances.
- C. Conclusions: Esterification of tobacco pectin is not effected by calcium hydroxide under the current treatment regime. Interaction of free pectin carboxyl groups does occur and may influence tobacco expansion.
- D. Reference:

Wooten, J., " $^{13}\text{C}$  CPMAS NMR of Calcium Hydroxide Treated Expanded Tobacco and Isolated Pectin," Memo to W. Hempfling and K. Shafer, November 27, 1990.

### III. MS ANALYSIS OF INORGANIC MAGNESIUM COMPOUNDS

- A. Objective: To explore the feasibility of in situ analysis of inorganic magnesium compounds by tandem mass spectrometry.
- B. Results: Fast atom bombardment and direct probe low voltage EI ionization using the JEOL SX 102/ SX 102 tandem mass spectrometer have been explored as methods for formation of characteristic ions from inorganic magnesium compounds. Fast atom bombardment ionization yielded some potentially informative ions.
- C. Conclusions: Experiments still are in progress.
- D. Plans: The potentially informative ions will be investigated by MS/MS. If this work yields satisfactory results, analysis of these compounds on paper will be pursued.
- E. Reference:  
Jensen, N., PM Notebook #8910, p. 80.

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